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Original Research Paper

Study the effect of dip in reaction temperature on thermal and electrical properties of ZnO nanoparticles

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ABSTRACT

The present study reports thermal and electrical properties of Zinc oxide (ZnO) nanoparticles prepared using co-precipitation route. Sudden dip in reaction temperature observed during the synthesis process created defects in the crystal lattice of ZnO which leads to reduction in crystallite size from 33 nm to 28 nm with increase in reaction temperature. This is confirmed by the X-ray diffraction studies. Thermal analysis of the samples shows better thermal stability for smaller nanoparticles. Temperature-dependent current-voltage characteristics of the ZnO samples show reduction in the conductivity and increase in dielectric constant with respect to rise in reaction temperature. Increase in dielectric constant with decrease in size of nanoparticles may be useful in the field of nanoelectronics like memory-storage devices, etc.

1. Introduction

Metal-oxide nanoparticles (MNPs) have drawn tremendous attention of researchers because of the reduced dimensions, superior optical and mechanical properties, better thermal stability and excellent transport properties [1,2]. The superior characteristics of these NPs are due to high surface to volume ratio and quantum confinement effect [3–5]. In nano dimensions, the morphological and electrical properties of the NPs are altered due change in the bandgap thus making them suitable for optoelectronics devices. Research on semiconductor nanocrystals has grown significantly in recent years owing to their novel optical, electrical and catalytic properties [4]. Amongst variety of MNPs reported till date, Zinc oxide (ZnO) NPs have proved to be one of the most versatile and functional nanomaterial because of their high biocompatibility [3,6], cost-effective and easy synthesis process, better control over shape and size, superior optical properties and better thermal stability [6,7]. ZnO is a II–VI semiconductor material with a wide band gap (3.37 eV) and the large exciton binding energy (60 meV) at room temperature [5,8]. Surplus techniques for synthesis of ZnO NPs are available which can be broadly categorized as chemical state (solid-state and wet-chemical routes), vapor state and biological methods [10–12]. Amongst them, chemical co-precipitation synthesis is the most cost-effective, reliable and environment-friendly technique and provides better control over the size and shape of the final product. Since the analysis of dielectric properties of pure ZnO NPs are not addressed properly in the literature, therefore, the authors have opted to make as detailed study the dielectric properties of ZnO NPs.

In the present work, the authors have adopted a novel synthesis method for the synthesis of ZnO NPs. Novelty of the present work is that for the first time effect of temperature dip is utilized in the co-precipitation method which results in reduction in the particle size of the obtained ZnO NPs with increase in reaction temperature. The reduction in size of the ZnO samples may be attributed to fast nucleation during the synthesis process [10]. The effect of dip in reaction temperature on the thermal and electrical properties of the samples was analyzed and discussed.

2. Methodology

Zinc acetate dihydrate ($Zn(CH_3COO)_2{\cdot}2H_2O$) and Sodium hydroxide (NaOH) of 99.95% purity were purchased from Sigma-Aldrich and used as-procurred. 0.1 M of $Zn(CH_3COO)_2{\cdot}2H_2O$ was added to 100 ml of double-distilled water (at 70 °C) in a sealed flask fitted with a burette and stirred at 700 rpm. 100 ml of equimolar Sodium hydroxide (at room temperature) was added

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Table 1
List of final ZnO samples (T1-T5) synthesised at different reaction temperatures.

S. no.	Sample	Precursor concentration		Molar ratio	Reaction temperature	Heating time
		Zinc acetate	NaOH			
1.	T1	0.1 M	0.1 M	1:1	70 °C	2 h
2.	T2	0.1 M	0.1 M	1:1	80 °C	2 h
3.	T3	0.1 M	0.1 M	1:1	90 °C	2 h
4.	T4	0.1 M	0.1 M	1:1	100 °C	2 h
5.	T5	0.1 M	0.1 M	1:1	110 °C	2 h

to the zinc solution and the mixture was stirred for 1 h at same temperature. The mixture was then refluxed at 70 °C for 1 h. The final mixture was cooled at room temperature for precipitation. The precipitate was then washed with methanol three times to remove the organic impurities and other unreacted compounds. The final precipitate was then dried in vacuum at room temperature to obtain the final product (Zinc oxide). Same procedure was followed to prepare Zinc oxide at different reaction temperatures (80 °C, 90 °C, 100 °C and 110 °C) keeping $Zn(CH_3COO)_2{\cdot}2H_2O$:NaOH molar ratio fixed at 1:1. The final ZnO powders (T1-T5) are listed in Table 1. The complete set of experiment was repeated twice to check and confirm the variation of particle size of the samples with respect to reaction temperature.

All the characterizations were performed at room temperature. The crystal structure and purity of the samples was characterized using X-ray diffractometer Rigaku MiniFlex II (1.54 Å Cu Kα radiation) in the range 20°–80° (2θ) at scan rate of 3°/min. Thermal properties were analyzed by Thermal Analyzer (Model: STA 449 F3; Make: NETZSCH (Germany)) in air atmosphere within the temperature range 30 °C/10.0(K/min)/300 °C. Electrical properties like electrical conductivity (σ), dielectric constant (ε′) and loss tangent (tan δ) were measured using the conventional parallel plate capacitor method using Impedance analyzer (Wenn-Kerr 6500B) for all the samples at four frequencies 1 kHz, 10 kHz, 100 kHz and 1 MHz in the above mentioned temperature range. Bias voltage was kept constant at 1 V and the temperature was controlled to an accuracy of ±0.5 °C. The area of the pellet in contact with the electrode is same as that of the electrode.

3. Results and discussion

3.1. X-Ray diffraction (XRD) analysis

Fig. 1(a) shows typical XRD patterns of the as-prepared ZnO samples. All the diffraction peaks in the obtained patterns matched well with the JCPDS file #80-0075 of the standard ZnO and can be indexed corresponding to the hexagonal reflections of the wurtzite ZnO. No characteristic peaks of the impurity were observed (see Fig. 2).

On comparing with standard JCPDS data, the diffraction peaks of the samples show broadening and a slight shift towards lower Bragg angle (2θ) which represents the reduction in crystallite size as well as presence of uniform strain [13,14]. The values of lattice parameters (a and c), crystallite size (calculated using Scherrer equation and W-H plot), lattice strain and strain along c-axis are calculated as discussed in our previous reported works [3,6] and

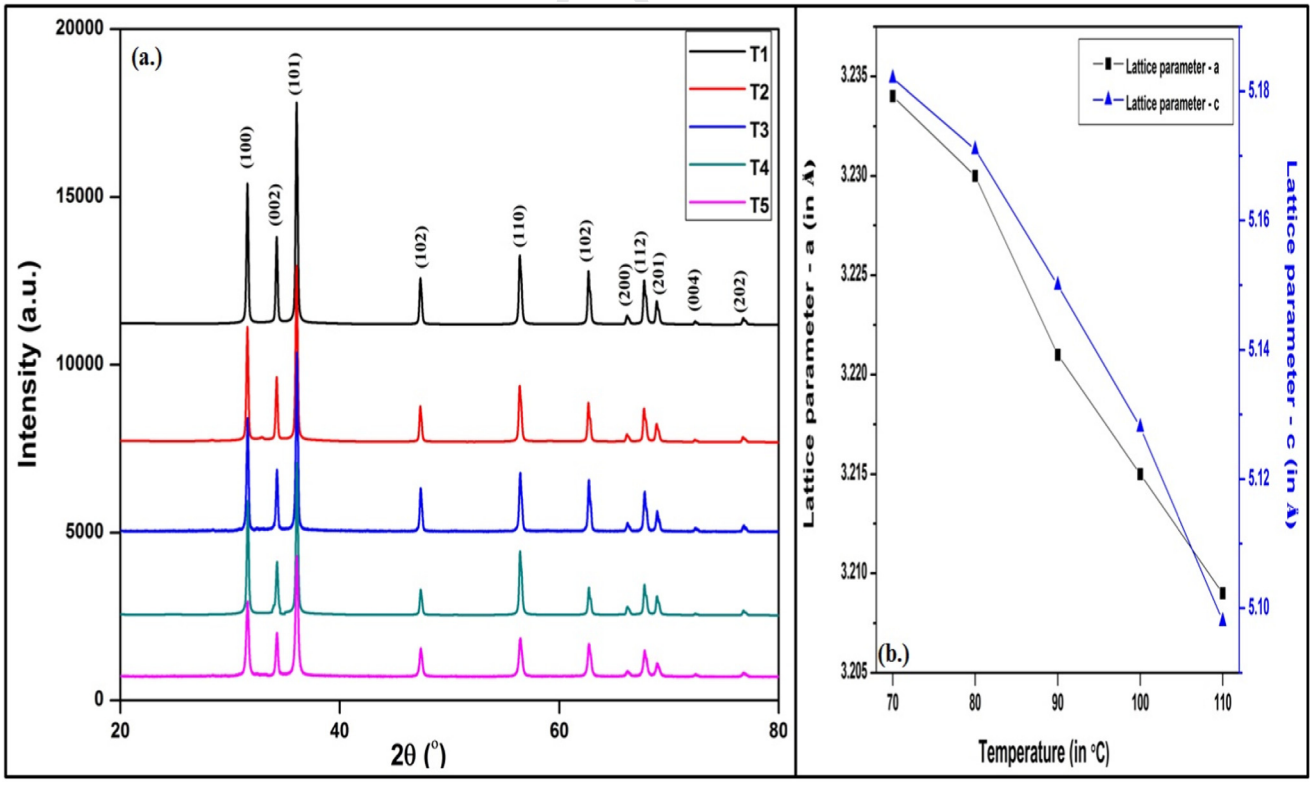

Fig. 1. (a) XRD pattern of ZnO samples prepared at different reaction temperatures; (b) Variation of lattice parameters (a and c) with reaction temperature.

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